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The report describes research performed by four Fellows who based their M.S. theses in Electrical or Materials Engineering on this effort. All work related to rare-earth permanent magnets and their applications in microwave or mm-wave devices. It prepared the candidates for employment in the permanent magnet-producing or the microwave tube industries.--The specific topics of the four sub-projects were:

1. Design Analysis of Permanent Magnet Field Sources; ~~Fellow: Anthony C. Morcos~~
2. Study of the Effects of Heat Treatment Variables on Microstructural, Physical and Magnetic Properties of Sm(Co,Fe,Cu,Zr)$_{7\frac{1}{2}}$ Alloys. ~~Fellow: Jeffrey L. Calvert~~
3. Interfacing Instruments for Magnetic Measurements to a Computer.-Fellow; ~~Gregory M. Umana~~ and
4. Characterization of Modified Nd-Fe-B Magnets for Application in Microwave Devices at Elevated Temperatures. ~~Fellow: Tom K. Tran~~

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GRADUATE FELLOWSHIPS IN THE FIELD OF HARD MAGNETIC MATERIALS

FINAL SUMMARY REPORT

DR. KARL J. STRNAT
F.M. TAIT PROFESSOR, ELECTRICAL
AND MATERIALS ENGINEERING

15 JULY 1989

U.S. ARMY RESEARCH OFFICE

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GRADUATE FELLOWSHIPS IN THE FIELD OF
HARD MAGNETIC MATERIALS

1. FOREWORD

This is the concluding report of a grant that provided funds for four graduate research fellowships in the general field of permanent magnet materials. The four fellows conducted their experimental research and analytical/design work under the tutelage of three professors in the Magnetics Laboratory of the University of Dayton. One fellow was awarded a master's degree in electrical engineering, one in materials engineering, the third will receive his MSEE degree on July 30, 1989. The fourth expects to defend his MSEE thesis later in 1989. Three of the fellows are now employed in industrial positions in which they are directly using the specialized knowledge and experiences gained in their thesis research: two with permanent magnet manufacturing firms and one with a producer of microwave tubes for military and space applications. The fourth fellow returned to the university after a work period with an electric utility company to complete his thesis.

All work done by the research fellows was part of a long-range project to develop permanent magnets based on rare earth-transition metal alloys. Objectives of this project are to better understand the physical-metallurgical mechanisms controlling the magnetic hardness and microstructure of sintered magnets, to improve and characterize the engineering properties of such magnets, and to develop criteria for their proper use in the design of microwave/millimeter-wave tubes and other critical devices. During the 4 1/2-year active lifetime of this grant, the specific problems changed as our program evolved and the general knowledge of rare-earth permanent magnets grew. We also tried to attune the research and thesis topics to the personal interests and perceived strengths of the individual students. Each fellow's work was guided by the one of three participating professors whose present research interests were closest to the fellow's topic.

While this grant financially supported the students, the group of involved faculty advisors--the Professors A.E. Ray (Metals and Ceramics), H.F. Mildrum (Electrical/Instrumentation Engineering) and K.J. Strnat (Electrical and Materials Engineering)--worked under a sequence of interrelated government research contracts which supported the advisors' research. As a consequence, each fellow's thesis subject was closely tied to one of these contracts, and his research results under his ARO-grant topic can generally not be strictly distinguished from the advisor's research under the related contract. These contracts/grants are identified below. All have the common practical objective of supporting the use of permanent magnets in electron-beam focusing structures such as traveling wave tubes, ubitrons or free-electron lasers. In these devices a high magnetic energy density is important for the sake of miniaturization, and a precise knowledge of the temperature variation of magnetic remanence and coercivity between -50° and +150°C, as well as an understanding of slow changes of the magnetic operating

flux with time of exposure at elevated temperatures ("magnetic aging") are important features for the device designer. The work of the different fellows aimed either at improving these materials properties, or at characterizing them, or at analyzing the device performance as a function of characteristic properties of the newly developed permanent magnets.

Here are the research contracts/grants with which the four graduate fellows interacted:

(a) US Army Contract No. DAAK20-84-K-0458, with the Army Electronics Technology and Devices Laboratory, 1984-1988. "Sintered Permanent Magnets Based On Nd-Fe-Co-B Alloys Having Improved Elevated Temperature Properties."

(b) US Army Contract No. DAAL03-87-K-0082, ARO, 1986-1990. "Metallurgical Processes in Multi-Component Rare Earth-Transition Metal Permanent Magnet Alloys."

(c) US Navy Contract No. N00014-87-K-2064, NRL, 1987-1989. "Development of Temperature Stabilized High-Performance Permanent Magnet Materials for Microwave Tube Applications."

The four thesis projects were related by the overall project goal, but they were in fact rather diverse in their specific objectives and methods. This makes it difficult to summarize them in a coherent way. They will therefore be discussed individually, in chronological sequence of their execution, as far as problem statements and results are concerned.

2. TABLE OF CONTENTS

3. TECHNICAL DISCUSSION OF THE FOUR FELLOWS' PROJECTS

3.1 Design Analysis of Permanent Magnet Field Sources

Fellow: Anthony C. Morcos
Degree: MSEE
Advisor: Dr. Karl J. Strnat

Period of full ARO stipend: 1 Jan.1985 - 31 Dec.1985, preceded by part-time support for preliminary studies from 1 July - 31 Dec. 1984.

3.1.1 Problem Statement:

To study the unique design possibilities for microwave tube structures and proposed new devices for the production of polarized millimeter-wave radiation made possible by the use of rare-earth permanent magnets. Specifically, to analyze magnetic field sources intended for use with devices for the production of circularly polarized electron radiation.--More generally, Mr. Morcos intended to prepare himself for professional work in the analysis of permanent-magnet field/force structures and the computer-aided design of such devices.

3.1.2 Summary of Important Results:

The analytical work for this thesis project was done with the cooperation of the US Army Electronics Technology and Devices Laboratory (ETDL), and partly at the ETD Laboratory facilities.

In general terms, this was a study of the stray magnetic fields, leakage flux, and end effects associated with various tube structures. The use of cladding and bucking magnets, and of soft-magnetic shielding to reduce the flux leakage (and thereby improve the performance of such magnetic circuits) were investigated. The ultimate goal of this project was the design of tube structures which channel the maximum amount of magnetic flux into the "useful" region of the tube while using a minimum amount of magnetic material.

Several specific magnetic circuits were considered. A dual-cavity klystron tube employing radially magnetized cladding magnets, first proposed by Neugebauer of GE, provides the basis upon which several later designs are based. Various ETDL modifications of this design also use cladding, as well as end magnets and bucking magnets, and are an attempt to reduce the overall size and weight of the tubes. Parametric studies were performed in order to determine the extent to which the leakage fields can be minimized in such structures.

Another important area of investigation was the design of permanent magnet structures for the extraction of circularly polarized radiation from electron beams, replacing the current-carrying windings with a permanent magnet arrangement. Dr. Klaus Halbach (Lawrence Berkeley Laboratory) presented a "pure CSEM (charge-sheet equivalent material) dipole" in a recent paper, and a clever

variation of this geometry should produce the desired effect. Another possible design, employing the cladding concept, was proposed by Dr. H.A. Leupold (US Army ETDL). The thesis topic involved the design and analysis of this type of device. The goal was the design of a permanent magnet arrangement producing the field configuration necessary for the extraction of circularly polarized radiation, using a minimum amount of magnetic material, and reducing leakage fields to an absolute minimum.

The Naval Research Laboratory designed a relativistic free-electron laser which produces circularly polarized millimeter-wave radiation. This is accomplished by means of a helical transverse magnetic field; i.e., the field orientation rotates continuously in the Φ-direction with displacement along the z-axis. The NRL planned to employ a bifilar solenoid to generate the necessary field configuration. It had to carry a current of 100 amperes to produce a 450 Oe field.

The primary concern of this thesis project was the design and analysis of a suitable permanent magnet field source which could replace the NRL's "power-hungry" solenoid and its bulky power supplies. Two candidate designs were presented, analyzed, and compared. Both are relatively efficient (low flux leakage) magnetic circuits in which thin segments, each producing a dipole field in the working air gap, are rotated relative to one another in order to produce the necessary helical magnetic field. Calculations showed that such permanent magnet devices can easily generate fields several times the 450 Oe presently generated with the NRL's bifilar solenoid. The effects of tightening the pitch of field rotation (which affects the wavelength of the output radiation) were investigated. Some practical and economical considerations and limitations inherent in the construction of these magnet structures were discussed.

Computers were used extensively in the analysis stages of this thesis project. The hardware employed was the ETDL's DEC PDP-11, their VAX 11-780, and the University of Dayton Magnetics Laboratory's IBM CS9000 laboratory computer. The A.O.S. Magnetic Analysis Program, a finite element computer code, helped in the analysis of complex structures by allowing for the solution of Maxwell's equations, and thus the computation of the flux density, within the devices.

3.1.3 Other Comments on the Fellow's Activities:

A.C. Morcos, spent the summer of 1985 working under Dr. Herbert Leupold at the Army ET&DL in Fort Monmouth, New Jersey on the design and analysis of the permanent magnet helical field source for use in free-electron lasers. Two papers were published concerning this project. (See Section 4A)

In November 1985 Mr. Morcos attended a short-course entitled "Microwave High-Power Tubes and Transmitters" at George Washington University in Washington, D.C. This course related closely to his thesis topic, and it served to provide him with a general

background in the area of high-power microwave and millimeter-wave devices. Mr. Morcos' primary concern has been the magnetic circuits employed in such tubes, but he consciously developed a broader knowledge in microwave technology through formal course work.

In November 1985, after writing his thesis, he returned to Fort Monmouth under invitational travel orders from the Army to complete work which had remained unfinished in the summer. Travel and living expenses for both visits to Ft. Monmouth were financed by the host laboratory, and no costs except the basic stipend were incurred by the fellowship grant fund.

Tony Morcos left the University in January, 1986, to work in the field of his graduate studies in the High Frequency Tube Dept. of the Hughes Aircraft Company's Electron Dynamics Div., Torrance, California.

3.2 A Study of the Effects of Heat Treatment Variables on Microstructural, Physical, and Magnetic Properties of $Sm(Co,Fe,Cu,Zr)_z$ Alloys

Fellow: Jeffrey L. Calvert
Degree: M.S. Materials Engineering
Advisor: Dr. Alden E. Ray

Period of full ARO stipend: 1 Jan.1986 - 31 Dec.1986, preceded by 2 years of part-time work as an undergraduate research technician in the same field, under an ARO contract.

3.2.1 Problem Statement:

This was primarily a metallurgical problem. The magnets of choice for traveling wave tubes, klystrons, free-electron lasers, wigglers and undulators are the rare-earth magnets based on the intermetallic compound $Sm_2(Co,Fe)_{17}$. These high-energy-density magnets have the best elevated temperature behavior and long-term flux stability, thanks to their high Curie temperature and oxidation resistance. However, their good coercivity suffers when more iron is introduced in the alloy and when the content of the crucial minor constituents, Cu and Zr, is reduced. Such compositional changes are desirable because they further increase the energy product of the magnet and make the alloy cheaper. It is thus important to understand the microstructural factors that influence the coercive force and learn how they might be controlled by proper heat treatments. This was the thrust of the thesis project.

Mr. Calvert was to work on a microstructural study of the development of coercivity in $Sm(Co,Fe,Cu,Zr)_z$ permanent magnet materials and the influence of processing parameters on the second quadrant of the hysteresis loop. The project involved designing a set of experiments that efficiently test the effects of specific variables in the heat treatment. Some specific goals of the

work were, first, to develop a fine-grain, single-phase and dense microstructure; second, to develop high coercivity by proper heat treatment; and third, to develop a "square" demagnetization curve in sintered magnets made from these alloys.

3.2.2. Summary of Important Results:

A study of the effects of heat treatment variables on $Sm(Co,Fe,Cu,Zr)_z$ alloys (z=8.513 and 8.285) was conducted. The sintering temperature and time, the sintering atmosphere, and the solid solution heat treatment (SSHT) temperature and time were varied at two levels. The effects of these variables on the grain size, the amount of Sm-rich second phase, the mass density, and the saturation magnetization were observed after the sintering and SSHT. The materials received a subsequent aging treatment consisting of an initial isothermal aging, varied at two levels, followed by a step aging. The final magnetic properties were measured, and the relationships between the magnetic properties and the microstructural properties were observed.

This study was initiated to determine the effects of the heat treatment variables on the grain size, and the effects of grain size on the final magnetic properties and on the squareness of the demagnetization curve. It was observed that the grain size varied from 25 µm to 65 µm with the heat treatment variables and levels chosen. The factors affecting the grain size, density, and final magnetic properties are discussed in the thesis, along with some recommendations for further experimentation.

3.2.3 Second Fellow's Subsequent Activities:

After his graduation in December 1986, Jeffrey Calvert accepted a position as Development Metallurgist with the Hoeganaes Corp. in Riverton, NJ, with responsibility for developing new production processes for Nd-Fe-B magnets. This well-established international powder-metallurgical firm recently added sintered Nd-Fe-B permanent magnets as a new product line. Calvert also helped to build up a quality control laboratory. Early in 1989 he moved to the magnet company I.G. Technologies, in Valparaiso, IN, as Manager of Process Control for their rare-earth magnet production plant.

3.3 Interfacing Instruments for Magnetic Measurements to a Digital Computer

Fellow: Gregory M. Umana
Degree Sought: M.S. - Electrical Engineering
Advisor: Dr. Karl J. Strnat
Lab. Supervisor: Prof. Herbert F. Mildrum

Period of ARO Stipend: 1 Jan.1987 - 28 Feb.1988, preceded by a period of related work under a US Army (ETDL) research contract.

3.3.1 Problem Statement:

The objective of the thesis project was to provide a means for characterizing permanent magnets more efficiently by use of a digital microcomputer while retaining the high accuracy and resolution provided by the analog measuring instruments previously used. A digital microcomputer, interfaced properly with magnetic measuring equipment, is an important time-saving tool for research facilities, like the University of Dayton's Magnetics Laboratory, and also for magnet manufacturing plants and industrial magnet users. Specifically, the improved capabilities for semi-automated measurement and more rapid data processing was required for subsequent research programs under US Army and Navy sponsorship aimed at improving and fully characterizing Nd-Fe-B-type permanent magnets.

The thesis will explain the basic methods of these magnetic measurements, discuss pertinent properties of permanent magnet materials, and the pre-existing instrumentation at the UD Magnetics Laboratory that was to be interfaced with the computer. The magnetic measuring methods addressed are those utilized in the following instruments: a hysteresigraph, a vibrating sample magnetometer (VSM), an oscillating specimen magnetometer (OSM), and open-circuit remanent flux (OCRF) pull-coil fixtures used in conjunction with an integrating digital voltmeter/fluxmeter (IDVM) for measuring temperature coefficients, irreversible and long-term aging losses.

The thesis project provides the hardware and software required to interface these systems with the Laboratory's previously purchased IBM 9000 Computer System. It was difficult to obtain hardware and software compatible with this uncommon system that is no longer produced. The software was written from scratch; the needed hardware, although it was difficult to locate, could be purchased. The main program language was Fortran. The data acquisition software was written in Assembler Code for the M-68000 microprocessor.

The interfacing of the microcomputer with each of the test systems permits the evaluation of a large number of permanent magnet samples in a short time and avoid errors due to operator fatigue. Typical information furnished consists of: (1) the salient magnetic properties of permanent magnets, such as remanent induction and coercive force, (2) magnetization and demagnetization curves, intrinsic (M vs. H) and "normal" (B vs. H), and (3) data to predict the temperature stability of permanent magnets.

3.3.2 Summary of Important Results:

The interfacing hardware and software was completed for the hysteresigraph, VSM and pull coil/IDVM systems. The satisfactory functioning of the three systems was demonstrated and procedures for the use by others were developed and documented. The Fellow also participated, with Prof. H.F. Mildrum, in an experimental project

under Army-ETDL sponsorship to study the properties of early commercial sintered Nd-Fe-B permanent magnets at elevated temperatures. The results have been published.--The systems and programs developed by Mr. Umana have recently been used extensively by others in this laboratory, and for other projects, both government and industry sponsored.

3.3.3 Other Comments on Fellow's Activities:

Upon completion of his project, in March, 1988, Greg Umana began working at the Magnequench Division (Delco-Remy) of General Motors in Anderson, Indiana. His initial assignment was to design specialized magnetic test equipment for quality assurance in permanent magnet production. We are happy that he could immediately put to use in the US magnet industry the specialized experience gained under this DoD fellowship.

However, the demands of his employment and personal circumstances have so far kept him from completing his thesis manuscript. It is now hoped that he will submit it at the end of summer, 1989, then defend his work and graduate in the academic fall term in December, 1989.

3.4 <u>Characterization of Modified Nd-Fe-B Magnets for Application in Microwave Devices at Elevated Temperatures</u>

Fellow: Tom Kern Tran
Degree: M.S. - Electrical Engineering
Advisor: Dr. Karl J. Strnat
Lab. Supervisor: Prof. Herbert F. Mildrum

Period of ARO Stipend: 1 January - 31 July 1988, preceded by a period of related work under a US Navy (NRL) research contract.

3.4.1. Problem Statement:

The objective of this project was to generate design information on the behavior of modified sintered Nd-Fe-B magnets over the typical operating temperature range of microwave tubes. It was part of a study of the utility of heavy-rare-earth substituents for improving the temperature coefficients and stability of such magnets. Specifically, the effects of holmium (Ho) substitutions in Nd-Fe-B, alone or in combination with cobalt (Co) were to be investigated.

Two methods of measurement were employed: a hysteresigraph was available for plotting intrinsic and normal demagnetization curves in closed circuit, and an induction pull-coil technique to measure the open-circuit remanent flux. Both instruments had been computer-interfaced by the earlier efforts of another ARO/DoD-Fellow. Hysteresis curve measurements are made on cube-shaped magnets; a special probe fixture enables the test samples to be heated or cooled in the desired temperature range. The temperature

functions of salient magnetic properties needed by design engineers can be determined from these curves, and the temperature coefficients of remanence and intrinsic coercive force calculated.

For the irreversible loss measurements, sets of axially magnetized cylindrical magnet samples of 0.25" diameter and three different lengths must be prepared, with permeance ratios $B/H \sim -2$, -1 and -0.5 in open circuit. The magnets are exposed at a specific temperature in air, cooled to room temperature, and the irreversible flux loss measured with a precision integrating digital voltmeter. The reversible loss and temperature coefficients of the operating-point flux values are obtained from similar flux measurements on free-standing samples, but made at elevated temperature, using a pyrex-and-quartz heater/pull-coil fixture.

Both the hysteresigraph and the pull-coil appartus are interfaced with an IBM-9000-System computer. All data were stored and later retrieved to make necessary calculations and to plot the results.

3.4.2 Summary of Important Results:

This thesis investigates the characteristic behavior of modified sintered Nd-Fe-B magnets over the temperature range of -50° to 150°C. Since Nd-Fe-B magnets have poor flux stability at elevated temperature, promising modifications of the basic ternary magnet alloy with Co, Dy and various other elements have previously been studied. In the project of which this thesis is a part, the effect of combined cobalt and holmium addition on the temperature behavior was investigated. Magnets of composition $(Nd_{1-x}Ho_x)(Fe_{0.92-y}Co_yB_{0.08})_{5.3}$ with three cobalt levels, y=0, 0.06 and 0.12, and with holmium substitutions for neodymium between 0 and x=0.25 were characterized. Demagnetization curves were measured between about -50°C and +150° to 200°C. Reversible temperature coefficients of remanence and intrinsic coercive force were derived from these. Irreversible flux losses on heating at elevated temperatures up to 175°C were determined.

The dependence of properties on the holmium content, x, the cobalt content, y, and the temperature is discussed and analyzed. Even though the remanence remains almost constant with the addition of cobalt, the energy product and temperature coefficients improve, particularly $\alpha(B_r)$. The loop squareness of the alloy with the highest Co content, y = 0.12, is very respectable. However, as was reported before for magnets without heavy-rare-earth additions, the coercivity decreases with increasing Co-content. Increasing Ho-content slightly raises the coercivity, but it cannot completely offset the $_MH_c$-loss due to cobalt addition. Ho substitution also leads to a decline of the energy product. It generally reduces (i.e., it improves) the temperature coefficients of remanence and coercivity, and it also improves the loop squareness. However these changes are small. It must be concluded that holmium additions are not a cost-effective way of achieving temperature compensation in sintered Nd-Fe-B based magnets with or without cobalt.

4. LIST OF PUBLICATIONS AND TECHNICAL REPORTS

4.1 Theses for the University of Dayton

a) Anthony C. Morcos, "The Measurement of Radial and Axial Forces between Permanent Magnets." Honors Thesis (special undergrad. project report, University of Dayton, School of Engineering, November 1984.

b) Anthony C. Morcos, "The Design and Analysis of a Permanent Magnet Field Source for the Production of Circularly Polarized Radiation via Free-Electron Lasers." M.S.E.E. Thesis, University of Dayton School of Engineering, November 1985.

c) Jeffrey L. Calvert, "A Study of the Effects of Heat Treatment Variables on Microstructural, Physical, and Magnetic Properties of $Sm(Co,Fe,Cu,Zr)_z$ Alloys." M.S.-Mat. Engr. Thesis, University of Dayton, School of Engineering, December 1986.

d) Tom K. Tran, "Characterization of Modified Nd-Fe-B Magnets for Application in Microwave Devices at Elevated Temperatures." M.S.E.E. Thesis, University of Dayton, School of Engineering, July 1989.

e) Gregory M. Umana, "Interfacing Instruments for Magnetic Measurements to a Digital Computer." Tentative title of an M.S.E.E. thesis still in preparation at the time of this writing. (July 1989)

4.2 Papers in Journals or Conference Proceedings Books

a) A.C. Morcos, K.J. Strnat and J.L. Calvert, "Measurement of Forces between Magnets in Passive Bearing Systems Employing Rare-Earth Magnets." Proceedings 8th International Workshop on Rare-Earth Permanent Magnets and Applications, pp. 162-177, May 1985.

b) Herbert A. Leupold and Anthony C. Morcos, "A Permanent Magnet Field Source for the Production of Circularly Polarized Radiation via Helical Free-Electron Lasers," Proceedings of the International Conference on Insertion Devices for Synchrotron Sources; SPIE Proceedings Vol. 582, Paper 582-15, Stanford, California, October 1985.

c) Anthony C. Morcos, Ernest Potenziani, and Herbert A. Leupold, "Permanent Magnet Structures for the Production of Transverse Helical Fields." IEEE Trans. Magnetics, vol. MAG-22, p. 1066, September 1986.

d) Herbert F. Mildrum and G.M. Umana, "Elevated Temperature Behavior of Sintered 'Nd-Fe-B Type' Magnets." IEEE Trans. Magnetics, vol. MAG-24, p. 1623, March 1988.

e) K.J. Strnat, H.F. Mildrum, Tom K. Tran and Y. Xiao, "Sintered Nd-Fe-B Magnets Modified With Holmium and Cobalt." Proceedings of the 10th International Workshop on Rare-Earth Magnets and Applications, Vol. 1, p. 523 May 1989.

5. PARTICIPATING SCIENTIFIC PERSONNEL AND DEGREES EARNED

a)	Patricia L. STUMPFF	(Withdrew from program in Oct. 1983)
b)	Anthony C. MORCOS	M.S. Electrical Engineering, Dec. 1985
c)	Jeffrey L. CALVERT	M.S. Materials Engineering, Dec. 1986
d)	Tom K. TRAN	M.S. Electrical Engineering, July 1989
e)	Gregory M. UMANA	Candidate for M.S. Electrical Engineering (1989)